DEVELOPMENT AND MODIFICATIONS OF BI-METALLIC NANOPARTICLES USING ION BEAMS TO TAILOR OPTICAL PROPERTIES FOR ADVANCED APPLICATIONS

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

to

Manav Rachna University

by

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It is the work that is expected to be carried out under the supervision of **Dr. Aditya Sharma and co-supervision of Dr. Jaiprakash and Dr. Fouran Singh**. Further, it is certified that short synopsis is within the plagiarism limit and copy of the same is attached as per University norms.

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ABSTRACT

Bimetallic nanoparticles (NPs) exhibit outstanding catalytic, magnetic, photonic properties. They can be of potential use in many applications. Development of these bimetallic NPs with tunable properties can be achieved through variation in composition, size, structure and shape. Using ion implantation technique, their composition can be easily varied. Thus, it enables tuning of their Surface Plasmon Resonance (SPR) property in a wide range in between those of the monometallic NPs. The structural changes from spherical to elongated shape can widen up this range further towards the longer wavelength. Swift heavy ions (SHI) irradiation can be employed to produce size growth, reduction and elongation of the metallic NPs embedded in silica matrix depending on the size of nanoparticles due to high electronic energy, S_e deposition in silica. Synthesizing alloy or core-shell structures can lead to drastically change in optical responses. We investigate two different Au based bimetallic nanoparticles in silica matrices: (i) miscible elements, Au and Ag, and (ii) immiscible elements, Au and Ni. Alloying of Au and Ag, it is possible to have a stable chemical stability and good optical response with tunable SPRs. Fusion of the magnetic, Ni and the optical element, Au in one single entity promises multi-functionality and potential applications. Rutherford Backscattering Spectrometry (RBS), X-ray diffraction (XRD), UV-VIS absorption spectroscopy, Transmission Electron Microscopy (TEM) and Superconducting Quantum Interference Device – Vibrating Sample Magnetometer (SQUID- VSM) will be employed to characterize the properties of the bimetallic nanoparticles.

Key words: Bimetallic nanoparticles, Surface Plasmon Resonance (SPR), Ion implantation, Core-shell, Swift heavy ion (SHI) irradiation

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1. Introduction

Bimetallic nanoparticles are of research interest because of their excellent optical, chemical, catalytic, magnetic properties and possible use in numerous applications. Particularly, their optical property, Surface Plasmon resonance (SPR) based plasmonic incorporated devices find potential applications for Bio sensing, solar cells, surface enhanced spectroscopy and optical switching. SPR occur when the electromagnetic field from the light impinges on the metallic nanoparticles (NPs) and the free conduction electrons of these metallic nanoparticles (NPs) collectively oscillate in resonance. A strong SPR absorption in visible spectrum can be produced by noble metal nanoparticles. Therefore, SPR properties of noble metal nanoparticles can be exploited for fabrication of biosensor. SPR peak shifts produced due to molecular interactions on the surface of NPs can be monitored to detect the biomolecules [1-3]. Development of the devices depends primarily on the properties of these metal nanoparticles which can be manipulated by carefully controlling their composition, structure, size and shape [1-4]. The tunability of SPR in a wide range is desired for many applications. SPR is tunable in a narrow range for monometallic NPs. Alloys can be made from two miscible elements. Their composition is one parameter which can be varied easily using sequential ion implantation technique. Therefore, SPR of alloy is tunable in a wide range in between those of the monometallic NPs. NPs synthesized in the form of nano rod (elongated shape) can widen up the range further towards the longer wavelength [5]. The elongated shaped NPs produce plasmon resonances of two bands. Their energy separation increases with increase in aspect ratio. Depending on the concentration of the constituent elemental metals, their chemical interaction as well as the synthesis technique employed, they can form alloy, hetero-structures and core shell structures [3-4]. Synthesizing core-shell structures lead to drastically change in optical responses due to changes in the structure and dielectric constant in core –shell boundary. The constituent elements of bimetallic NPs have different thermal expansion coefficients. Thus, they can form hollow shells structure upon cooling after annealing at high temperature. One of the constituent elements can also be enriched on the nanoparticle surface due to segregation towards surface [6]. This process may lead to the formation of core–shell structure of the nanoclusters.

Swift heavy ion (SHI) irradiation is known to create ion tracks in silica matrices due to high electronic energy, S_e deposition. Thus, upon SHI irradiation, the embedded bimetallic NPs grow, elongate as

well as reduce in size inside silica matrices [1, 7]. Besides, there can be occurrences of phase-decomposition as well as re-precipitation of constituent elements in silica matrices.

Surface Plasmon Resonance (SPR) properties of metal NPs are employed widely as a mechanism to assess and retrieve the absorption of nano sized materials and biomaterial on the surface of metal nanoparticles. The SPR based biosensors utilize refractive index changes to detect biomolecules. The refractive index of metal NPs increases with the binding of the biomolecules on their surface. It results in variation in surface plasmon waves. The measured change in refractive index in real time provides information about the biomolecules without any time-delay [2, 8-10].

We aim to investigate two different Au based bimetallic nanoparticles in silica matrices: (i) miscible elements, Au and Ag, and (ii) immiscible elements, Au and Ni. Further, focus is on the applications of the synthesized bimetallic NPs as sensor for biomolecule or glucose. In the first case, Silver has an intense SPR but it is chemically unstable, but opposite is true for gold. Alloying these two metals, it is possible to have a stable chemical stability and good optical response. Both Au and Ag have fcc crystal structure and their lattice constants are very close to each other (4.078 Å for Au; 4.086 Å for Ag). According to their phase diagram and Hume-Rothery rules, Au and Ag can form alloy [11]. In the second case, the plasmonic properties of Au nanoparticlest are tunable [1]. Since fusion of the magnetic element, Nickel and optical elements, Au, in one single entity promises multi-functionality and potential applications, so a great interest to fabricate nanoparticles containing Au and Ni. The RBS, XRD, UV-VIS absorption spectroscopy, TEM and SQUID- VSM will be employed to characterize the properties of these bimetallic nanoparticles. The synthesized bimetallic nanoparticles will be utilized for fabrication of optical biosensors based on their optical property, SPR for the detection of biomolecules or glucose.

2. Literature Review

Bimetallic nanoparticles can be synthesized by various methods like atom beam co-sputtering deposition, so-gel and sequential ion implantation [4, 12-15]. The practical application of these nanocomposites strongly depends on the composition, size, shape and structure of the constituent metal nanoparticles. Miscibility of the two constituent elements play important role for alloy formation. However, it is not very stringent for nanoparticles since they have high surface area over volume [16].

M. Novakovića et al., Xuan Meng et al. and Francesco Gonella groups synthesized bimetallic AuAg alloy nanoclusters using sequential ion implantation technique, while S. Link et al. and several groups synthesized through chemical routes. They reported the tunability of the position of the surface plasmon resonance (SPR) band by just varying composition in the synthesized bimetallic AuAg nanoclusters [4, 12, 15-24]. Similar results were also obtained by T.S. Anderson group in AuCu alloys and they further observed formation of Cu shell with Ag core and their SPR position lies in between those of pure elements [13]. D. Rioux group calculated SPR positions of AuAg alloy nanoparticles of arbitrary compositions using an analytic model. They found SPR positions tunable and the results are in close agreement with the experimentally observed values for the AuAg alloy thin films prepared by co-evaporation method [25].

When two metallic species are implanted sequentially in dielectric matrix, it can form alloy, mixed clusters or core-shell structure as shown in figure 1. There is possibility of forming non-equilibrium phase during the synthesis and atoms are not at their thermodynamically prescribed position. It is necessary to anneal them to accelerate the inter diffusion of atoms that promote the formation of an alloy. Post implantation thermal treatment also leads to separation of the constituent components or oxidation often, instead of alloying [18, 26-27].

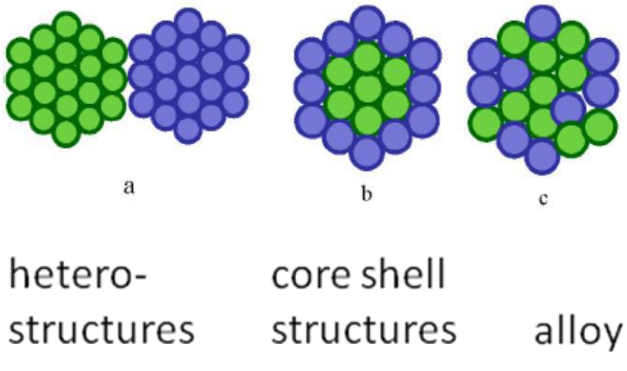

Figure 1. Bimetallic nanoparticles structures a) hetero structure, b) core-shell structure and c) alloy

Francesco Gonella observed Ag enrichment and its size growth in the cluster after annealing of as implanted AuAg alloy irrespective of the annealing environment (reducing/oxidizing) [17]. Thus, the annealing process offers a way to synthesize nanocomposites with wide range of optical properties.

Mostly, the researches so far focused on the synthesization of alloy nanoparticles using two miscible metals (AuAg, AuCu). But, there exists limited studies of alloy NPs which are prepared from the immiscible metals (AuNi) [28]. It will be interesting to determine their miscibility gaps in nano scale

regime. Zaneta Swiatkowska-Warkocka group manufactured AuNi alloy NPs successfully by means of the pulsed laser irradiation technique [29]. The alloy thus formed shows soft ferromagnetic properties at room temperature with a small coercivity (34 Oe) and small remnant magnetization (0.1 emu/g). This property of generating a strong magnetic signal using a small applied magnetic field is desirable for numerous applications.

Several reports showed NP size growth upon SHI irradiation, if they are smaller than ion track size; otherwise, they prolate along the direction of the ion beam [4]. Giancarlo Rizza et al. showed swift heavy ion (SHI) irradiation produced shape change of AuAg alloy NPs into elongated nanorods and nanowires in silica matrix [29]. Mishra group also observed SHI (90MeV Ni ion) irradiation induced Au NPs size growth [30].

Surface Plasmon Resonance (SPR) properties of bimetallic naoparticles have potential to utilize for lebel free biosensor for detection of the blood sugar and different biomolecules. The monitoring and management of blood glucose level are important for maintaining the health of diabetes patient. Several group reported direct detection of biomolecule/glucose using a surface plasmon resonance (SPR) biosensor in real-time based on the refractive index-changes due to binding of immobilized biomolecules on the surface of the metal NPs [2, 8-10].

3. Description of Topic

Noble metal nanoparticles (Au, Ag and Cu) embedded in dielectric matrices are of research interest due to their interesting optical properties in the UV-Visible regime. They show strong absorption called surface Plasmon resonance absorption. It occurs due to coherent free electron oscillation of metallic nanoparticles in the presence of electromagnetic field. Figure 2 is schematic diagram illustrating the light wave induced excitation of dipolar surface plasmon on a nanoparticle surface. SPR is observed only when the size of the particle is less than the wavelength of the incident light. The optical property depends also on the particles size since their band gap energy increases with decrease in size. The optical responses are directly proportional to the free electron density and their dielectric function.

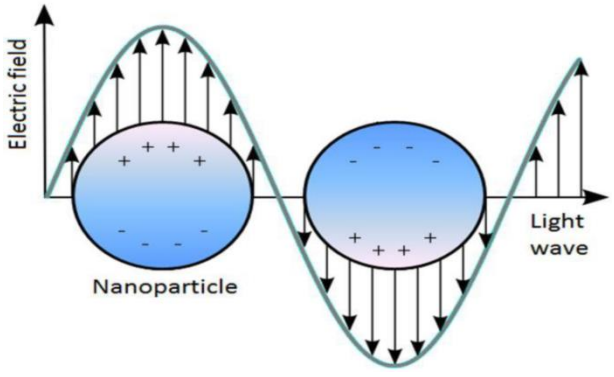

Figure 2. Schematic diagram illustrating light wave induced excitation of dipolar surface plasmon on a nanoparticle surface

For the metal nanoparticles of sizes lesser than 20 nm (quasi static regime), the extinction cross section is significantly contributed by the dipole oscillations (L=1) only. In presence of light wave with the angular frequency, ω, the optical absorption coefficient α of the metal nanoparticles embedded in dielectric medium of refractive index, n is [6]

$$\propto (cm^{-1}) = \frac{18\pi V n^3 \varepsilon_2}{\lambda((\varepsilon_1+2n^2)^2+\varepsilon_2{}^2)}$$

where $\varepsilon_1(\omega)$ and $\varepsilon_2(\omega)$ are the real and imaginary parts of metal dielectric function. V is the metal nanoparticle volume fraction. The maximum absorption (SPR) occurs if $\varepsilon_1+2n^2=0$ and ε_2 is small [6]. The dielectric function $\varepsilon(\omega)$ of alkalis is controlled largely by transitions of the free electrons within the conduction band. However, in case of noble metals, additionally, there is a considerable contribution of interband transitions from the core level electrons. Thus, the resonance is hybridization of joint actions of both the conduction and d-band electrons. The contribution of the electrons from the interband transitions to the susceptibility is given by $\chi^{IB}= \chi_1{}^{IB}+i\ \chi_2{}^{IB}$. The direct dissipation energy expressed by $\chi_2{}^{IB}$ is large for frequencies of interband transitions, whereas, $\chi_1{}^{IB}$ is significant for smaller frequency. $\chi_1{}^{IB}$ is replaced often by the polarization of ion core, χ^{core}, an averaged frequency independent value [6]. Thus, considering all the optical properties of materials, the complex dielectric function all over the UV-Visible region is

$$\varepsilon(\omega) = 1 + \chi\mathrm{IB}(\omega) + \chi\mathrm{DS}(\omega)$$

Where $\chi^{DS}(\omega)$ is Drude-Sommerfeld susceptibility for the free electrons.

For development of new optically and magnetically advanced material in nano scale regime, the underlying properties which play roles in their optical studies must be studied. The physicochemical properties of materials depend on the composition as well as their distributions inside the nanocomposite. Mie's theory can be employed to calculate the optical properties of alloy if their dielectric function is known [1]. The dielectric function of the AgAu alloy incorporating the weighted average of their composition is given by [5]

$$\varepsilon_{\text{alloy}}(\omega) = X_{ag}\varepsilon_{ag}(\omega) + (1 - X_{ag})\varepsilon_{au}(\omega)$$

Where, x_{ag} denotes the percentage of Silver metal. $\varepsilon_{ag}(\omega)$ and $\varepsilon_{au}(\omega)$ are the dielectric constants of corresponding bulk silver and gold metals.

The threshold excitation energies for Au (5d→6s) and Ag (4d →5s) are close to 3.9eV and 1.8eV respectively. These electronic transitions couple strongly with their free electrons responses resulting in the difference in their dielectric functions, Ɛ . Thus, the absorption spectra of core–shell structure can have two peaks. The strong coupling of the excited Surface Plasmon polariton modes of the core and shell via the interfaces leads to the complex optical absorption spectra [6].

The absorption cross section for the ellipsoidal particles in the quasi-static regime is given as [5]

$$б_{abs} = \frac{V}{3c}\epsilon_s^{3/2}\sum_{i=1}^{3}(Y_i + 1)^2 \quad \frac{\omega\epsilon_2}{(\epsilon_1 + Y_i\,\varepsilon_s)^2}$$

Where, V is the metal particle volume and c is the speed of light in a vacuum. $\varepsilon_m(\omega)$ and $\varepsilon_s(\omega)$ are dielectric constants of metal nanocluster and embedding material. ε_1 and ε_2 are the real and complex dielectric constants of the metal particle. Y_i is the geometrical polarization factor with three principal axes, i=a, b, c. The resonance occurs when $\varepsilon_m(\omega) + Y_i\varepsilon_s(\omega) = 0$. In the frequency range, visible to near-IR, for noble metals, ε_2 is small and $d\varepsilon_2/d\omega$ is negligible.

AuNi alloy nanoparticles can have both the optical and magnetic properties. Thus, the embedded AuNi alloy in dielectric matrices can offer the properties of superparamagnetic, enhanced coercivity, the hysteresis loop shift and large magneto transport [23]. Therefore it is interesting to investigate their optical and magnetic properties in correlation with variation in metal composition, thermal annealing and SHI irradiation.

4. Objectives of Research

1) Synthesize gold based bimetallic nanoparticles in quartz matrices by sequential ion implantation method (a) miscible elements, Au and Ag (b) immiscible elements, Au and Ni.

   Therefore, we will be preparing $Au_{1-x}Ag_x$ (x=0 to 1) and $Au_{1-x}Ni_x$ (x=0 to 1) etc.

2) Engineering of the nanoparticles in terms of composition ratio, size, shape and structures through sequential ion implantation, thermal annealing as well as swift heavy ion irradiation.

3) Characterize the bimetallic nanoparticles thus formed using XRD, UV-visible absorption spectroscopy. Photoluminescence spectroscopy. X-ray absorption spectroscopy, TEM with elemental analysis, RBS etc.

4) Investigate the composition, structural, optical and magnetic properties of the synthesized bimetallic nanoparticles

5) Look for potential applications of the synthesized nanoparticles in Bio-sensing or Glucose sensing.

5. Methodology to be adopted

Sequential ion implantation method will be employed for the synthesis of gold based bimetallic nanoparticles in quartz matrices. The ion energies will be chosen to ensure their maximum overlap in almost same projected depth range. It is pictorially shown in figure 3. Using SRIM (stopping and range of ions in matter) code, the ion energies are simulated. SRIM calculations are shown in figure 4. These samples will be subjected to activation by thermal annealing. The highly energized ions delivered by 15 UD Pelletron facility at IUAC, New Delhi will be employed for swift heavy ion irradiation. The elemental distribution of the synthesized bimetallic NPs inside the dielectric matrices will be determined. The synthesized NPs will be characterized for their optical, structure, composition and magnetic properties using various techniques.

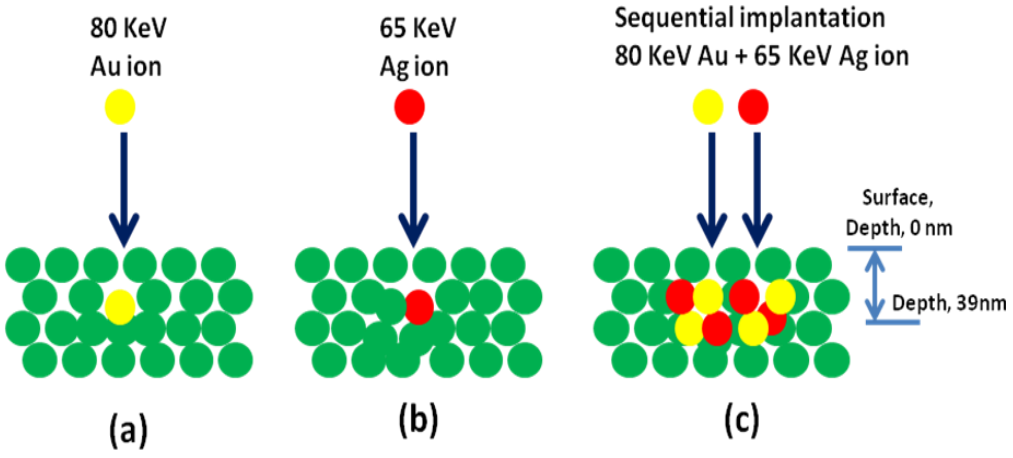

Figure 3. a) 80KeV Au^- ion implantation in quartz at a projected depth of 39nm , b) 65 KeV Ag^- ion implantation in quartz in almost same depth, c) Sequential ion implantation of 80KeV Au^- and 65 Ag^- ion showing Au and Ag element particle distribution overlap in almost same projected depth range

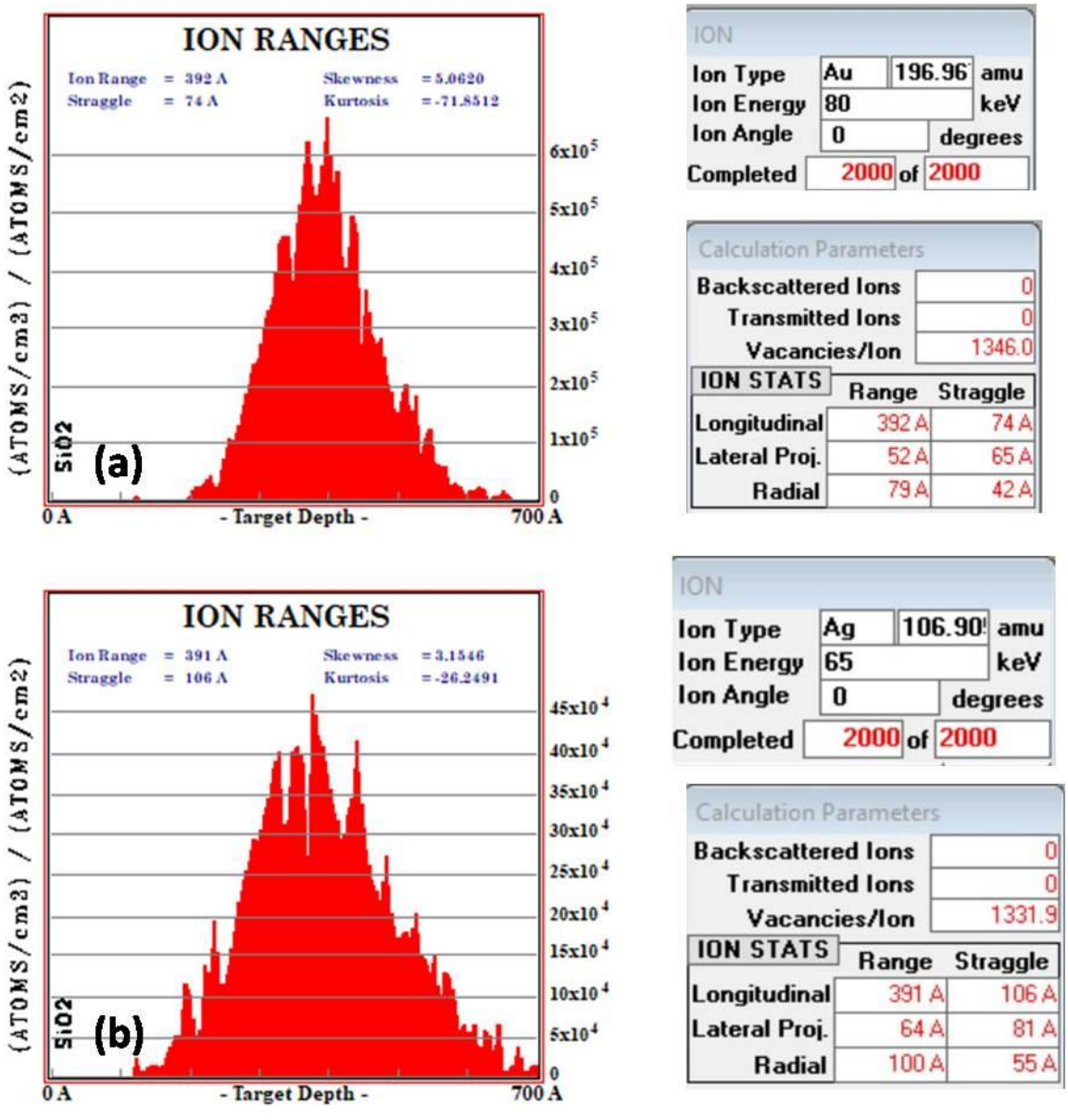

Fig. 4. SRIM simulated depth profile of (a) Au when 80 KeV Au^- implanted in Quartz along with its calculated parameters , (b) Ag when 65KeV Ag^- implanted in Quartz along with its calculated parameters.

Experimental Plan

Sequential Ion implantations

Sequential ion implantation of (i) 80keV Au^- and 65 keV Ag^- are required for synthesis of AuAg alloy nanoclusters and (ii) 80keV Au and 44keV Ni for that of AuNi alloy/core-shell nanoclusters with variables fluences in the range of 1e15 to 3e16 ions/ cm^2.

AuAg alloy in SiO_2:

Table 1. SRIM calculations for 80KeV Au^- and 65KeV Ag^- ion in Quartz

Ion species	Ion Energy E	projected range r	longitudinal straggling Δr	lateral straggling
Au	80keV	392A	74A	65A
Ag	65keV	391A	106A	81A

AuNi alloy/core-shell nanoparticles in SiO_2:

Table 2. SRIM calculations for 80KeV Au^- and 44KeV Ni^- ion in Quartz

Ion species	Ion Energy E	projected range r	longitudinal straggling Δr	lateral straggling
Au	80keV	392A	74A	65A
Ni	44keV	383A	135A	106A

Thermal annealing studies: This will be done mainly to control the size and growth of nanoparticles. This will be carried out both isochronical (at a particular time, different annealing temperature range 600° C -900° C) and isothermal (at a particular temperature, different annealing time) in air and inert environment to investigate diffusion of the constituent elements for the formation alloy and/or core shell nanoparticles.

Swift Heavy Ion Irradiation studies:

Swift heavy ion beam irradiation of the implanted samples will be carried out with different energy and variable ion fluencies. Ni and Ag ion beam of energy 120 MeV with a fluence varying from 10^{11} to 10^{14} in 1 cm^2 are required for the proposed work. This work will be done at IUAC, New Delhi. The irradiation of films will be done using 15 UD Pelletron facilities at IUAC, New Delhi. For ion track formation, the threshold electronic energy deposition in silica is 2 keV/nm [3]. Energy loss details of 100MeV Ag ions in silica are shown in table 3.

Table 3. SRIM calculations for 100MeV Ag ion irradiation in Silicia

Ion	energy MeV	Electronic energy loss Se keV/nm	Nuclear energy loss Sn eV/nm	Projectile Range Rp Mm	Substrate
Ag	100	11.72	60.32	14.60	Silica

Facilities required

The as implanted, annealed and swift heavy ion irradiated films will be characterized for their optical, composition, structural and magnetic properties using various characterization tools.

Compositional studies by RBS: This will be studied by 1.7 MV PARAS facility at IUAC,Delhi..

Structural Studies: This will be studied by using XRD and Cross-sectional TEM.

Optical Studies: The optical properties will be studied by using **UV-Visible spectroscopy** and **Photoluminescence spectroscopy**. The work will be carried out at IUAC, New Delhi.

X-ray absorption spectroscopy: For the determination of valence state of elements. The work will be done at Pohang Synchrotron Accelerator facility, South Korea,

Transmission electron microscopy: For the study of surface morphology of the Nano-structures and elemental analysis in Energy dispersive X-ray (EDX) analysis mode.

Magnetic Studies: The magnetic property measurement will be performed using Superconducting quantum interference device (SQUID)/Vibrating Sample Magnetometer (VSM).

6. Expected outcome of the research

The bimetallic nanoparticles with desired plasmonic properties may be synthesized for a particular application through changes in compositions, structure, shape and size. Moreover, the promising and futuristic application of this research is the Bio-sensing or Glucose sensing.

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Title: Development and modification of Li-metallic nanoparticles using ion beams to tailor optical properties for advanced applications.

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DEVELOPMENT AND MODIFICATIONS OF BI-METALLIC NANOPARTICLES USING ION BEAMS TO TAILOR OPTICAL PROPERTIES FOR ADVANCED APPLICATIONS

by Kshetrimayum Devarani Devi

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DEVELOPMENT AND MODIFICATIONS OF BI-METALLIC NANOPARTICLES USING ION BEAMS TO TAILOR OPTICAL PROPERTIES FOR ADVANCED APPLICATIONS

SHORT SYNOPSIS

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DEVELOPMENT AND MODIFICATIONS OF BI-METALLIC NANOPARTICLES USING ION BEAMS TO TAILOR OPTICAL PROPERTIES FOR ADVANCED APPLICATIONS

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